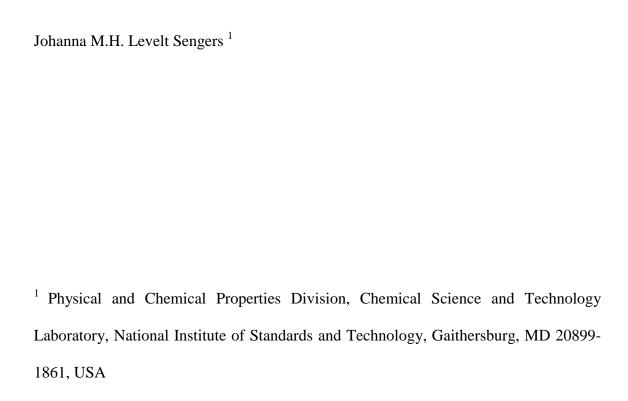
The Laboratory Founded by Van der Waals



ABSTRACT

During the past century, the Van der Waals Laboratory at the University of Amsterdam

has been the principal provider of reliable fluid property data over large ranges of

pressure and temperature. This paper describes the history of the laboratory, starting in

1898 when funding for it was obtained. In the early period, under Van der Waals and

Kohnstamm, the high-pressure direction was chosen, and the first PVT and phase

equilibria data were published. The main focus of this paper is the Michels period, from

1921-1960. In this period, the laboratory acquired its own building, and assumed a

unique position in the world because of its highly accurate thermodynamic, transport

and other property measurements in fluids at high pressures. In the 1950s, a second

laboratory was built by Michels at the University of Maryland, per request of the U.S.

Navy. Under Trappeniers, 1961-1987, the laboratory incorporated new techniques, such

as NMR, undertook a major expansion of the pressure range, and extended its interest to

phase transitions in molecular solids. The position of the Van der Waals Laboratory in

the world of high-pressure science is highlighted.

KEY WORDS:

High pressure; history; NMR; phase equilibria; PVT data; thermal conductivity;

viscosity

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1. INTRODUCTION

During the past century, the Van der Waals Laboratory of the University of Amsterdam has been a major provider of accurate thermophysical property data at high pressures. The organizers of the 14th Symposium on Thermophysical Properties considered it desirable to highlight the history of this laborastory at a time when it is undergoing major transformation, which includes termination of much of its traditional work.

In this paper, such a history is presented. In addition to presenting facts, dates and outputs, the paper will show the motivation for the undertakings. What did the founder have in mind, what models did he and his successors have, why were the directions chosen the way they were, what were the major achievements, and what was the impact? The paper is written in four sections, the first of which (Sec.2) describes the early period of Van der Waals and Kohnstamm, 1898 to 1920. Sections 3 and 4 concern the Michels period, 1920-1960, and Section 5 the Trappeniers years, 1961-1987. The main emphasis is on the Michels period, in which the laboratory carved out its work area, acquired its own building, built its reputation, and became known world-wide.

2. THE VAN DER WAALS FUND

For a proper understanding of the origin of the Van der Waals Laboratory, it is necessary to consider the circumstances of thermodynamics in the Netherlands in the late 1890s [1]. J.D. Van der Waals (1837-1923) had his major achievements well behind him: the equation of state, the theorem of corresponding states, the theory of

mixtures, and the theory of capillarity. His reputation was solidly grounded, and the experimental study of phase separations in mixtures was spreading all around him. His close friend H. Kamerlingh Onnes (1853-1926), had built a famous physics laboratory at the University of Leiden, and he was well on his way towards the liquefaction of helium. He had tested Van der Waals's theory of mixtures in glass vessels at pressures up to approximately 100 bar. The Chemistry Department in Amsterdam, under the leadership of H.W. Bakhuis Roozeboom (1854-1907), was furiously studying phase equilibria in liquids and solids in Cailletet tubes, vertical glass capillaries sealed at the top and immersed in mercury at the bottom. By injecting mercury, phase transitions could be observed, and crude volumetric data obtained at pressures up to about 100 bar. In France, E.H. Amagat was a pioneer in high-pressure studies. He was able to generate pressures up to 3000 bar, and for calibrating his manometers he had installed a mercury column in a deep mine shaft. Also, he had modified the Cailletet tube, enclosing it in an autoclave, and providing it with platinum contacts at intervals along the tube. This way, *PVT* data could be obtained up to high pressure.

While this experimental activity was going on all around him, Van der Waals had no facilities of his own. This was increasingly felt to be embarrassing, and thus, at the 25th anniversary of Van der Waals's 1873 thesis, colleagues and friends established the Van der Waals Fund. At first several institutions contributed, while, later, individuals paid a yearly subscription fee of five Dutch guilders. The history of this fund can be found in Ref. [2]. The stated purpose of the Fund was "to supply monetary support for about the next ten years for experimental research related to the theories put forward by the eminent scientist and to be conducted in Amsterdam under the supervision of the founder of the theory."

It must not have been easy for Van der Waals, as the Director of the Physics Laboratory, and heavily loaded with teaching, to get an experimental laboratory off the ground and moving. Fortunately, in the 1890s he was supervising a capable Ph.D. student, Ph.A. Kohnstamm (1875-1951), who took an interest in the laboratory. The direction was chosen to be high pressure. This was an obvious choice, in view of the pressure limitations experienced by Kamerlingh Onnes and by Bakhuis Roozeboom. In 1904, a large grant from the Van der Waals Fund was used to buy a Schaeffer and Budenberg press, autoclave, and several dead-weight gages. The upper range was 5000 bar. The laboratory was a 7 m by 7 m room in the Physics Lab, plus a small corridor that served as a machine shop. The first publications of the Van der Waals Fund began around 1910. At that time, the Belgian J. Timmermans, of later fame as an expert in liquid mixtures, was a guest at the laboratory, and one of the earliest papers by Kohnstamm and Timmermans was on liquid miscibility up to 3000 bar [3].

Around 1915, a new development is noted. Kohnstamm and Walstra developed an Amagat piezometer with platinum contacts, suitable for pressures up to 3000 bar. They measured *PVT* data for hydrogen around room temperature. This is the beginning of a *PVT* measurement method which the Van der Waals Laboratory would practice throughout the 20th century.

The Bakhuis Roozeboom student F.E.C. Scheffer (1883-1954) worked as an assistant at the laboratory. He studied 3-phase equilibria in Cailletet tubes. Scheffer later became a Professor at the Technical University of Delft. At Delft, studies of phase equilibria by means of Scheffer's method persist to the present day.

From 1915 to 1922, no publications resulted from the Van der Waals Fund.

Although the Netherlands was not involved in the first World War, students did have to

serve in the military. Kohnstamm's interest began to shift from experimental physics to paedagogics, to which he would devote the later part of his life. Worst, however, was the general belief that Van der Waals's work on molecular science was outdated. The available students flocked towards the rising star, the Amsterdam physics professor Pieter Zeeman (1865-1946), who became a Nobel laureate as early as 1902, not yet 40 years old, and eight years before Van der Waals was thus honored.

Two events are worth noting. In 1911, an instrument maker trained by Kamerlingh Onnes joined the laboratory. His name was J. Ph. Wassenaar. And A.M.J.F. Michels (1891-1969), a high-school mathematics teacher, began his math and science studies at the University of Amsterdam.

3. HISTORY OF THE MICHELS YEARS: 1920-1961

From WWI to 1930. Michels soon became associated with Kohnstamm, but his studies were interrupted by his military service. Around 1919, he resumed his studies and laboratory work in Amsterdam, while remaining a part-time high-school teacher. In the early 1920s, he began to put his considerable energy, and also a considerable part of his own earnings, into rebuilding the moribund laboratory. He made a major improvement in the design of the bottom-loaded differential pressure gage [4], and began to build these for the various pressure ranges, up to the maximum of 3000 bar. He quickly sensed that the lab could not be put on a solid footing without proper funding for his first priority: a pressure calibration facility.

Westertoren project. Unlike the larger European nations, the Netherlands had never had a standards laboratory. Thus, Michels had no convenient way of getting the

dead-weight gages of his design calibrated, resulting in a wide gap between "precision" and "accuracy" of pressure measurement. While temporarily working for Kamerlingh Onnes one day a week, Michels convinced the Nobel laureate of the necessity of a pressure calibration facility. In 1925, Kamerlingh Onnes embarked on a spirited fundraising drive advertised in the newspapers. The drive yielded a considerable sum for the Van der Waals Fund. Several Dutch companies then constructing pressure equipment did contribute. Michels used the funding for one of his more colorful projects. Following the example of Amagat, he would erect a tall mercury column. For lack of mine shafts in a city build on mud, Michels decided to use one of Amsterdam's architectural assets: a tall church spire, the 17th - century Westertoren. For years hence, this tower would house a 27.5 m tall mercury column, the only pressure standard in the world. As an open column, it could be used to calibrate gages up to the 300 bar range. Used differentially, it could be stepped up to the maximum pressure for which differential piston gages were constructed in the lab: 3000 bar.

New directions. Michels obtained his Ph.D, with Kohnstamm in 1924. His thesis topic was the accurate measurement of PV isotherms. In 1928, Kohnstamm took a leave of absence, and Michels obtained a full-time position as a lecturer at the University of Amsterdam. High school teaching days were over. The new course Michels set for the laboratory was:

- A substantial increase in accuracy of physical variable measurements.
- Large pressure and temperature ranges
- De-facto de-emphasis of phase equilibria studies
- Emphasis on molecular science
- A broad spectrum of physical properties of interest.

The entrepreneur. In the late 1920s, Michels reputation as an expert in high pressure came to the attention of Imperial Chemical Industries (ICI) who were then engaged in exploring pressure as a medium for new chemistry. They hired Michels as a high-pressure consultant. Company engineers would be sent to Amsterdam for training, and for obtaining advanced degrees. The company was unwilling to fund Michels through the University administration. A creative legal solution was found: the Boyle Foundation was established to receive industry support for instrumentation of the Van der Waals Laboratory. Through this Foundation, the laboratory quickly became one of the best equipped in the world. ICI was working on the polymerization of ethylene. The story is that in the early 1930s, in the course of PV measurements on ethylene at the Van der Waals Laboratory, the piezometer lost pressure. When the glass blower cut the piezometer open, it contained a sticky residue that could not be removed with chromic acid: the first sample of polyethylene. Polyethylene is said to have won the second World War: its low dielectric loss at high frequencies enabled development of cmwavelength radar by the British, who were then able to monitor the launching of planes and missiles from Germany.

The pressure-generating capabilities became prototypes for the design of highpressure chemical plants in various countries. Also, collaborative ventures resulted with
Dutch companies for constructing the necessary equipment. After the second World
War, one such Dutch Company, 't Hart, began to sell Michels-design pressure
equipment to the U.S. Navy, to American universities, and to companies around the
world. As a University lector, Michels was breaking new ground. Although consulting
with industry was common at the Technical University in Delft, at venerable academic
institutions such as the University of Amsterdam, such practices were unheard of.

In 1930, Kohnstamm retired and Michels succeeded him. His position remained that of a university lecturer, and it would not be until 1939 that the university considered him worthy of a professorship. By that time, Michels had earned a solid reputation abroad, and had found the means to build a first-rate high-pressure laboratory, with no more than lukewarm support from the university.

A new building. By 1930, the laboratory had outgrown its decrepit space in the Physics Laboratory, and Michels started planning for a new facility. The University was in the process of designing buildings to house the geology and the chemistry departments. Between the two buildings, room for an additional wing was available, and Michels did not waste any time claiming it. The new facility was occupied in 1935 and remained in use through the mid-1960s. The 4-story building had a suite of temperature-controlled vibration-free basement rooms, and a total of three floors of experimental rooms filled of pressure generating and measuring equipment. Emulating Kamerlingh Onnes's instrument makers school, Michels and Wassenaar designed a fully equipped machine shop, in which all pressure equipment needed by the laboratory was to be constructed by a group of several dozen trained machinists and apprentices. The building had a glass shop, a chemical preparation and purification laboratory, a hazards lab for pressure testing, and, later, an air liquefaction facility. It housed a library filled with the major journals, supplied by Michels' personal subscriptions, while the meager university contribution was used for books only. A standards-quality metrology laboratory was available for calibration of platinum thermometers, precision length measurement, and precision weight calibration of the 25-kg weights carried by the dead-weight gages. The new facility was undoubtedly the best-equipped highpressure laboratory in the world.

Meetinstituut. In 1947, Michels established a nonprofit foundation for calibration and precision measurement in industry. It was funded in part through an industrial consortium, in part by Government. Meetinstituut specialized in non-routine precision length measurements and pressure calibration for industry.

A second Van der Waals Laboratory. After the second world war, the Office of Naval Research and the Naval Ordnance Laboratory of the American Navy, much concerned about possible lags of American high-pressure science, approached Michels, with the request to build a copy of the Van der Waals Laboratory in the United States. Michels accepted this challenge, which made it necessary for him to spend several months a year in the U.S.A. for a number of years. The new facility was built at the University of Maryland in College Park. At groundbreaking in 1951, the first shovel of dirt was dug by the physicist J.D. Van der Waals, Jr., the only son of Van der Waals. The facility was opened in 1954. Presently, it is part of the Institute of Physical Science and Technology at the University of Maryland, specializes in theoretical statistical physics, and houses experimental light scattering facilities.

A consequence of the U.S. connection was a steady stream of American visitors to the Van der Waals Laboratory. They originated from U.S. Navy laboratories as well as from universities in order to receive training in high-pressure techniques, to obtain advanced degrees, or to prepare themselves for work at the new facility in College Park, MD. The Van der Waals Laboratory thus assumed an international atmosphere long before the globalization of science penetrated other academic institutions in the Netherlands.

4. WORK IN THE MICHELS YEARS

The Van der Waals Laboratory became a unique source of reliable thermophysical property data for fluids by combining a large pressure range, a high level of experimental accuracy, a wide scope of properties studied, and a generally well deserved reputation for reliability of its work. An overview follows of the most significant work. Since full referencing of over 300 publications and Ph.D. theses is clearly impossible, the author has severely and arbitrarily limited referencing. Virtually all of the experimental data on thermodynamic and transport properties have found their way into existing data bases, in which the references can be found.

PVT data for one-component fluids above 0 °C. Throughout the Michels years and beyond, the measurement was pursued of PV isotherms of gases in Amagat-type glass piezometers enclosed in autoclaves. Here the typical range was from 0 °C to 150 °C in temperature, and up to 3000 bar in pressure, the range originally chosen by Amagat. The target accuracy was one part in 10 000 in both pressure and density. Due to the accumulation of error in the "stepping-up" during calibration with respect to the mercury column, this target was not reached at the higher pressures. Nevertheless, the final uncertainty in the density, involving both pressure uncertainty and volume calibration uncertainties, was usually well below 1 part in 1000 in much of the range. The sensitivity of the temperature measurement was 1 mK, and the temperature gradients in the piezometer probably no larger than 0.01 K, at least for the lower half of the temperature range. In the Michels years, the laboratory built its own platinum thermometers, and calibrated them in a water triple-point cell of Michels's own invention in 1927 [5], long before such practice became internationally accepted. For

the rest of the temperature range, the thermometers were calibrated on the absolute temperature scale by extrapolating the *PV* data to zero density. It was believed that this could be done with an uncertainty of the order of 0.01 K. Table I contains a list of fluids studied during the Michels years and beyond, together with the year of publication of each of the experimental data sets.

Table I. Fluids studied in glass piezometers above 0 °C

Substance	Period	Substance	Period
nitrogen	1928, 1934	argon	1949
hydrogen	1915, 1932, 1941	methyl fluoride	1952
carbon dioxide	1930, 1935	propene	1953
carbon dioxide	1937	oxygen	1954
ethene	1936, 1942, 1976	xenon	1954
methane	1935, 1979	neon	1960
deuterium	1941	krypton	1964
helium	1941		

Although it is impossible to describe individual publications here, an exception may be made for carbon dioxide. The data extended Andrews's measurements in large measure, with unsurpassed accuracy. The 1937 paper [6], which investigated the critical region in great detail, contained the Ph.D. research of Michels's wife, C. Michels-Veraart, whose Ph.D. adviser was J.D. van der Waals, Jr., the son of Van der Waals.

In general, the publication of the experimental data was followed by a calculation of thermodynamic properties derived from the *PVT* data by numerical and graphical differentiation and integration. Human computational experts, employed by the Laboratory and outfitted with hand-cranked (later: electrically powered) calculators, thought nothing of doing least-squares fits to *PVT* data by means of up to sixth-degree polynomials in density. A full calculation of thermodynamic properties over the entire experimental range might take such an expert as long as a full year.

These numerical representations of the experimental data became the principal source of experimental second and third virial coefficients. The virials, in turn, yielded accurate information on intermolecular forces, as envisioned by the Founder. The heat capacity C_v derived from the data in the supercritical regime of fluids such as carbon dioxide and ethane showed a substantial increase. We now know that this was an indication of a weak divergence, whose measurement by Voronel and coworkers in 1961 initiated the rebirth of interest in criticality of fluids.

PVTx data for mixtures. At various times, such data were obtained in the same apparatus, for mixtures at one or a few fixed compositions. We mention the system methane-ethane, of interest to the gas industry (1939), ammonia-nitrogen-hydrogen (1949, 1951), related to ammonia synthesis at the Dutch State Mines (DSM), and air (1955).

Phase equilibria in mixtures. In the Michels years, phase equilibria studies played a surprisingly small role. The methanol-hydrogen system was studied in 1953, and three ammonia systems, that with nitrogen and hydrogen, with krypton, and with argon, were studied between 1959 and 1961.

Vapor pressures and PVT data below 0 °C. After the second World War, a beginning was made with an expansion of the temperature range. For this application, a steel piezometer was built, which was housed in a cryostat filled with a stirred, low melting point organic fluid such as isopentane, and connected by a fine capillary to a differential pressure gage, maintained at room temperature. This was not an absolute instrument: existing PVT data at 0 °C and above were used to calculate the fill density, and the amount of gas present in the differential gage. Using liquid nitrogen as a coolant, temperatures as low as - 170 °C were reached. The pressure range was up to 1000 bar. The target accuracy was again 1 part in 10 000 in pressure and density, but some degradation occurred due to the indirect way of obtaining the density, and due to decrease of the sensitivity of the differential membrane in case the fluid studied was in a range of low compressibility.

In the early 1950s, the apparatus was used for measuring vapor pressures of argon, krypton, carbon monoxide and nitrogen. Then, *PVT* data were obtained for air, argon [7], hydrogen and deuterium.

Transport properties: viscosity. The decision to include transport property measurements in the program of the laboratory was made by Michels at a fairly early stage. The first industrial associate from ICI, R.O. Gibson, obtained his Ph. D. in Amsterdam on viscosity measurements in nitrogen up to 1000 bar [8]. The apparatus was a glass capillary of carefully calibrated diameter. It was attached to a reservoir at the bottom. The reservoir was provided with four platinum contacts at different heights. The capillary-reservoir assemblage was mounted in parallel to a wider glass tube, and the system was enclosed in a pressure vessel. All volumes between the platinum contacts were calibrated using known PVT relations for the gas. After pressurizing the

gas by pumping mercury into the bottom of the viscometer, the mercury would rise quickly in the wide tube, thus pressurizing the gas in the reservoir below the capillary, and establishing a pressure differential across the capillary. The gas would then transpire through the capillary upwards from the reservoir. The time it took for the mercury to travel from one platinum contact to the next one up was a measure of the fluid viscosity. The range of the apparatus was from 0 °C to 75 °C, and up to 2000 bar. The density of the fluid followed from the measured average pressure and temperature. For fluids not in regions of high compressibility, where the pressure gradient does not affect the density unduly, the method yielded viscosities believed to be accurate to within a few tenths of a percent. For pressurized gases, this was at the time the best instrument available in the world. After the second World War, viscosities were measured for hydrogen and deuterium (1953), carbon dioxide (1962), neon (1964), and krypton (1965).

Transport properties: thermal conductivity. Rather late in the Michels years, the decision was made to build a thermal conductivity apparatus. The intended range was 0 °C to 75 °C, at pressures up to 2600 bar. The apparatus was of the parallel-plate type, with adjustable spacings down to less than 1 mm, and a guard ring ensuring a linear profile of the gradient. The top plate was heated, so that, at least in fluids of low compressibility, a stationary state was obtained free from convection. Near a critical point, the tendency towards convection increases enormously, and it took several rounds of reconstruction of the apparatus to eliminate convection to within 0.1 K from the critical point in carbon dioxide. This effort paid off: in 1962, a critical anomaly was reported in the thermal conductivity [9] of a type not forecast by the Van Hove theory then believed to be correct. This discovery led to the modern theory of critical

dynamics, the mode-mode coupling theory, which has since been verified for a multitude of long-range dynamic fluctuation phenomena.

Other properties: refractive index. In the mid-1930s, an interferometer was built suitable for refractive index measurements between 0 °C and 75 °C, up to 2600 bar. It was used for the fluids carbon dioxide (1937), ethylene (1947), nitrogen (1947) and argon (1949). For these fluids, the equation of state had been measured earlier. This was the first time the weak density dependence of the Lorentz-Lorenz function was studied in detail over a large density range.

Other properties: dielectric constant. The work on the dielectric constant began in the early 1930s. The experimental range was from 0 °C to 150 °C, at pressures up to 3000 bar. In the 1930s, data were obtained for nitrogen, carbon dioxide [10], and hydrogen. After the second World War, data were taken for argon. The Van der Waals laboratory thus was the first to explore the density dependence of the dielectric constant and to demonstrate the weak variation of the Clausius-Mossotti relation over large ranges of density.

Other properties: absorption spectroscopy. In the mid-1950s, the laboratory branched out in the direction of studying optical phenomena induced by pressure. At that time, the theory of pressure broadening of spectral lines, and of pressure-induced absorption, were being developed at the Institute for Theoretical Physics in Amsterdam and elsewhere. The time was ripe for experimental verification of this new branch of molecular physics: the influence of Van der Waals forces on optical absorption [11]. As usual, the range of the apparatus was chosen to be from from 0 °C to 150 °C, at pressures up to 3000 bar. Initially, the effect of various pressurized gases on a mercury

absorption line was measured. Later, evidence was found for weakly bound mercuryinert gas compounds [12].

Other properties: various. To conclude the overview of the Michels years, I mention a body of measurements of the melting curves of the noble gases [13], and an interesting study of the adsorption of nitrogen on alumina at high pressures [14].

3. NEW DIRECTIONS IN THE TRAPPENIERS YEARS

Nuclear magnetic resonance. N.J.Trappeniers, who had obtained his Ph. D. with J. Timmermans, one of the early guests at the Van der Waals Laboratory in the Kohnstamm years, succeeded Michels in 1961. A Professor of Physical Chemistry in Groningen, he brought to Amsterdam a large group of Groningen students well versed in the technique of nuclear magnetic resonance. Large magnets began to appear in Amsterdam, and a new and unique capability was forged the Van der Waals Laboratory, that of NMR at high pressures, and, later, at low temperatures. The design capabilities of the shop were stretched to the limit, and creative solutions were found for contrary requirements: close spacing of magnetic poles; access for probes of the magnetic field; heavy walls for pressure containment; and space for thermal insulation. The need for cryogenic work well below that liquid nitrogen range was felt early, and facilities for hydrogen liquefaction were developed, soon to be followed by the use of liquid helium. Much of the NMR work was reviewed by K.O. Prins [15]. The early measurements were those of self-diffusion in several fluids. These data showed the absence of a critical anomaly in this coefficient [16]. Then, the emphasis shifted to the study of phase transitions in molecular solids, such as ethylene, ammonium chloride, and methane. The latter work, in a range of 1.5 K - 4 K, became a spectacular success when a number of high-pressure phases were detected which owed their existence to quantum effects [17].

Expansion of the pressure range. Early in the Trappeniers years, the artificial Amagat barrier of 3000 bar was surmounted. In part, the studies of solid phase transitions required much higher pressures, with less of a demand for ultra-precise pressure measurements. On the other hand, the quest for high pressures was fueled by the studies of phase equilibria in mixtures, carried out by J.A. Schouten [18]. The expansion of the pressure range occurred in two steps. More or less conventional means were used to reach pressures up to 15 kbar. To confine and characterize gaseous mixtures under such conditions is by no means conventional, however, and the Van der Waals Laboratory was one of the first to acquire this capability. The next major step was the use of the diamond anvil cell, and spectacular results began to appear in the literature on fluid-solid phase equilibria obtaned in systems with gas-gas phase separation. This paper will not describe these results, since they will be covered in the companion paper by J. A. Schouten. As a result of the new activities, the Van der Waals Laboratory quickly recovered its reputation as a premier high-pressure The establishment and flourishing of the international AIRAPT laboratory. conferences, initiated by Trappeniers in collaboration with Vodar in France, and E.U. Franck in Germany, is testimony to the clout and reputation of the laboratory.

4. CONCLUSION

Two forces propelled the Van der Waals Laboratory to its unique place in the world of thermophysical property measurement: the strong Dutch tradition of molecular science, and the singular energy, vision, engineering skill and entrepreneurship of its Director, Michels.

This is not the place to comment on the fate of the Van der Waals Laboratory after it took leave of its last director, Trappeniers, in 1987. Forces well beyond the control of the laboratory engulfed the physical sciences at the University of Amsterdam, and mergers and reorganizations resulted.

Let us remember that when Michels began his career at the laboratory in 1920, he found it near death. It took one visionary to revive the laboratory and launch it on a long and strikingly successful course spanning most of the 20th century. We may well gain hope when listening to the young voices now sounding from the Van der Waals-Zeeman laboratory and represented at this Symposium

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